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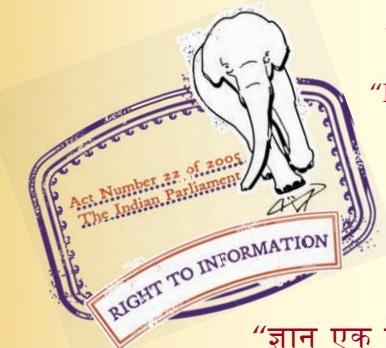
“Step Out From the Old to the New”

IS 11603 (1986) : Ammonium thiocyanate, photographic grade
[CHD 5: Electroplating Chemicals and Photographic Materials]

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Bhartṛhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”



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Indian Standard
SPECIFICATION FOR
AMMONIUM THIOCYANATE,
PHOTOGRAPHIC GRADE

UDC 771·7 : 661·862·38



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR AMMONIUM THIOCYANATE, PHOTOGRAPHIC GRADE

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Indian Standard
**SPECIFICATION FOR
AMMONIUM THIOCYANATE,
PHOTOGRAPHIC GRADE**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 February 1986, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 Ammonium thiocyanate is used as a photographic chemical and analytical reagent.

0.3 In the preparation of this standard, assistance has been derived from ISO 3622-1976 'Photographic grade ammonium thiocyanate — Specification', issued by the International Organization for Standardization (ISO).

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ammonium thiocyanate (NH_4SCN), photographic grade (molecular mass 76.1) used in the processing of sensitized photographic materials.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless, deliquescent crystals.

*Rules for rounding off numerical values (*revised*).

2.2 Appearance of Solution — An aqueous solution (10 g/100 ml) shall be clear and free from sediments.

2.3 The material shall comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

**TABLE 1 REQUIREMENTS FOR AMMONIUM THIOCYANATE,
PHOTOGRAPHIC GRADE**

| SL No. | CHARACTERISTIC | REQUIREMENT | METHOD OF TEST (REF TO CL NO. IN APPENDIX A) |
|-----------|--|-------------|--|
| (1) | (2) | (3) | (4) |
| i) | Assay (as $\text{NH}_4 \text{ SCN}$), percent by mass, <i>Min</i> | 97·0 | A-2 |
| ii) | pH of 5 percent aqueous solution | 4·5-6·0 | A-3 |
| iii) | Chlorides (as $\text{NH}_4 \text{ Cl}$), percent by mass, <i>Max</i> | 0·1 | A-4 |
| iv) | Sulphates [as $(\text{NH}_4)_2 \text{ SO}_4$], percent by mass, <i>Max</i> | 0·06 | A-5 |
| v) | Residue on ignition, percent by mass, <i>Max</i> | 0·10 | A-6 |
| vi) | Heavy metals (as Pb), ppm, <i>Max</i> | 20 | A-7 |
| vii) | Iron (as Fe), ppm, <i>Max</i> | 5 | A-8 |
| viii) | Sulphur compounds (as S), ppm, <i>Max</i> | 50 | A-9 |

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable airtight containers as agreed to between the purchaser and the supplier.

3.2 Marking — Each container shall be securely closed after filling and marked legibly and indelibly with the following information:

- a) Name of the material;
- b) Mass of the material in the container;
- c) Name of the manufacturer and/or his recognized trade-mark, if any; and
- d) Lot or batch number and the date of manufacture.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative sample of the material shall be drawn as prescribed in Appendix B.

A P P E N D I X A *(Clause 2.3)*

METHODS OF TEST FOR AMMONIUM THIOCYANATE, PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see IS : 1070-1977**) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. ASSAY

A-2.1 Reagents

A-2.1.1 *Dilute Nitric Acid Solution* — 1 : 9.

A-2.1.2 *Ammonium Iron (III) Sulphate Solution* [$\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$] — 5 percent (*m/v*).

A-2.1.3 *Standard Silver Nitrate Solution* — 0.1 N.

A-2.1.4 *Standard Ammonium Thiocyanate Solution* — 0.1 N.

*Specification for water for general laboratory use (*second revision*).

A-2.2 Procedure — Weigh accurately about 0·3 g of the sample and dissolve in 50 ml of water. Add 5 ml of nitric acid solution followed by 50 ml of 0·1 N silver nitrate. Shake well, add 2 ml of ammonium iron (III) sulphate solution and titrate the excess of silver nitrate with 0·1 N ammonium thiocyanate solution.

A-2.3 Calculation

$$\text{Assay (as } \text{NH}_4\text{SCN}), \text{ percent by mass} = \frac{7.612 (50 F_1 - F_2 V)}{M}$$

where

V = volume in ml of the ammonium thiocyanate solution used for the titration,

F_1 = factor of 0·1 N silver nitrate,

F_2 = factor of 0·1 N ammonium thiocyanate, and

M = mass in g of the material taken for the test.

A-3. pH OF THE SOLUTION

A-3.1 Apparatus

A-3.1.1 pH Meter — with glass and calomel electrodes.

A-3.2 Procedure — Prepare a five percent aqueous solution of the material in a 100-ml beaker. Measure the *pH* of the solution using a *pH* meter.

A-4. TEST FOR CHLORIDES

A-4.1 Reagents

A-4.1.1 Sodium Hydroxide — solid.

A-4.1.2 Dilute Nitric Acid Solution — 1 : 9.

A-4.1.3 Hydrogen Peroxide Solution — approximately 7 percent.

A-4.1.4 Standard Chloride Solution — 0·01 g of ammonium chloride dissolved in 1 000 ml of the nitric acid solution (1 ml = 0·01 mg).

A-4.1.5 Silver Nitrate Solution — 10 percent.

A-4.2 Apparatus

A-4.2.1 Nessler Cylinders — 50-ml capacity.

A-4.3 Procedure — Weigh accurately about 1 g of the sample and dissolve in 30 ml of hydrogen peroxide solution in a conical flask. Add 1 g of sodium hydroxide and rotate until the vigorous reaction ceases.

Then add a further 30 ml of hydrogen peroxide solution and boil for 2 min. Cool and dilute to 100 ml with water. Transfer 10 ml aliquots of this test solution and of the standard chloride solution to separate Nessler cylinders and treat each solution as follows. Add 10 ml of nitric acid solution, dilute to 50 ml and then add 1 ml of silver nitrate solution and mix well.

A-4.3.1 Compare, in the Nessler cylinders, the turbidities produced after 5 min in the test and control solutions.

A-4.3.2 The sample shall be taken as conforming to this requirement when the turbidity in the test solution shall be not greater than that produced in the control solution.

A-5. TEST FOR SULPHATES

A-5.1 Reagents

A-5.1.1 *Dilute Hydrochloric Acid Solution* — 1 : 9.

A-5.1.2 *Barium Chloride Solution* — 100 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) dissolved in 1 000 ml of water.

A-5.1.3 *Standard Sulphate Solution* — A soluble salt dissolved in water to give a solution containing 0·1 mg of sulphate [$(\text{SO}_4)^{2-}$].

A-5.2 Apparatus

A-5.2.1 *Nessler Cylinders* — 50-ml capacity.

A-5.3 Procedure — Weigh accurately about 0·9 g of the sample and dissolve in 30 ml of water in a Nessler cylinder. Transfer 4 ml of standard sulphate solution to the other Nessler cylinder. Add 0·5 ml of the hydrochloric acid solution and 1 ml of barium chloride solution to each, dilute to 50 ml, mix well and let it stand for 15 minutes.

A-5.3.1 Compare, in the Nessler cylinders, the turbidities produced in the test and control solutions.

A-5.3.2 The sample shall be taken as conforming to this requirement when the turbidity produced in the test solution shall be not greater than that produced in the control solution.

A-6. DETERMINATION OF RESIDUE AFTER IGNITION

A-6.1 Apparatus

A-6.1.1 *Platinum Crucible*

A-6.1.2 *Muffle Furnace* — capable of being controlled at $600 \pm 50^\circ\text{C}$.

A-6.2 Procedure — Weigh accurately about 5 g of the sample into the previously weighed platinum crucible. Heat at 300°C in a fume cupboard until volatilization is complete and then ignite the residue in the furnace controlled at 600 ± 50°C, for 4 h. Cool in a desiccator and weigh to the nearest 1 mg.

A-6.3 Calculation

$$\text{Residue after ignition,} \quad \text{Percent by mass} = \frac{M_3 - M_1}{M_2 - M_1} \times 100$$

where

M_1 = mass in g of the crucible,

M_2 = mass in g of the crucible and test portion, and

M_3 = mass in g of the crucible and residue.

A-7. TEST FOR HEAVY METALS

A-7.1 Reagents

A-7.1.1 Dilute Hydrochloric Acid Solution — (1 : 99).

A-7.1.2 Dilute Ammonia Solution — 1 : 9.

A-7.1.3 Standard Lead Solution — a soluble lead salt dissolved in water to give a solution containing 0·01 mg of lead per ml.

A-7.1.4 Water — saturated at room temperature with hydrogen sulphide.

A-7.1.5 p-Nitrophenol Indicator Solution — 0·25 percent.

A-7.2 Apparatus

A-7.2.1 Nessler Cylinders — 50-ml capacity.

A-7.3 Procedure — Weigh accurately about 2 g of the sample and dissolve in 20 ml of water in a Nessler cylinder. Also transfer 4 ml of the standard lead solution to the other Nessler cylinder. To each, add 1 drop of the *p*-nitrophenol indicator solution followed by the ammonia solution, drop by drop, until the solution turns yellow. Add the hydrochloric acid solution, drop by drop until the solution becomes colourless and then add 0·5 ml in excess. Finally add 5 ml of the hydrogen sulphide water, dilute to 50 ml and mix well.

A-7.3.1 Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

A-7.3.2 The sample shall be taken as conforming to this requirement when the colour produced in the test solution shall be not greater than that produced in the control solution.

A-8. TEST FOR IRON

A-8.1 Reagents

A-8.1.1 Acetate Buffer Solution, pH 5.0 — Dissolve 23 g of anhydrous sodium acetate in 58 ml of 2 N acetic acid and dilute to 1 000 ml. Adjust the final pH to 5.0 ± 0.1 with glacial acetic acid of 100 g/l sodium hydroxide solution.

A-8.1.2 Standard Iron Solution — Dissolve a soluble iron (III) salt in water to give a solution containing 0.01 mg of iron (III) per ml.

A-8.1.3 1, 10-Phenanthroline Reagent Solution — Thoroughly mix equal volumes of a 1 g/l aqueous solution of 1, 10-phenanthroline, a 100 g/l aqueous solution of hydroxylammonium chloride and the acetate buffer solution.

A-8.2 Apparatus

A-8.2.1 Nessler Cylinders — 50-ml capacity.

A-8.3 Procedure — Weigh accurately about 10 g of the sample and dissolve in 20 ml of water in a Nessler cylinder. Transfer 5 ml of the standard iron solution to the other Nessler cylinder. Add 10 ml of the 1, 10-phenanthroline reagent solution to each, mix and allow to stand for 10 minutes. Dilute each to 50 ml and mix well.

A-8.3.1 Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

A-8.3.2 The sample shall be taken as conforming to the requirement when the colour produced in the test solution shall be not greater than that produced in the control solution.

A-9. TEST FOR SULPHUR COMPOUNDS PRECIPITATED BY AMMONIACAL SILVER NITRATE

A-9.1 Reagents

A-9.1.1 Ammonia Solution — relative density 0.91.

A-9.1.2 Sodium Sulphide Nonahydrate Solution — 1 percent (m/v).

A-9.1.3 Silver Nitrate Solution — 10 percent (m/v).

A-9.1.4 Standard Silver Nitrate Solution — 0.001 N.

A-9.2 Apparatus

A-9.2.1 Nessler Cylinders — 50-ml capacity.

A-9.3 Procedure — Weigh accurately about 1 g of the sample and dissolve in 25 ml of water. Add this solution to a mixture of 20 ml of ammonia solution and 2 ml of silver nitrate solution. Also prepare a control solution by adding a few drops of the sodium sulphide solution to a mixture of 25 ml of water, 20 ml of ammonia solution and 3 ml of standard silver nitrate solution. Heat both solutions in a boiling water bath for 15 minutes, cool, transfer to the Nessler cylinders and dilute to 50 ml.

A-9.3.1 Compare, in the Nessler cylinders, the colours produced in the test and control solutions.

A-9.3.2 The sample shall be taken as conforming to the requirement when the colour produced in the test solution shall be not greater than produced in the control solution.

NOTE — Dispose off all test solutions and rinse apparatus used immediately. Explosive compounds may be formed on standing.

A P P E N D I X B

(Clause 4.1)

SAMPLING OF AMMONIUM THIOCYANATE, PHOTOGRAPHIC GRADE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 For general requirements of sampling, the methods given in IS : 8883 (Part 1)-1978* may be followed.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same grade drawn from a single batch of processing shall constitute a lot. If a consignment is declared or known to consist of different batches of processing, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Tests for determining the conformity of the lot to the requirements of the specification shall be done on each lot separately.

B-2.2 The number of containers to be selected shall depend upon the size of the lot and shall be in accordance with Table 2.

B-2.3 The containers shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS : 4905-1968† may be followed.

*Methods of sampling chemicals and chemical products: Part 1 General requirements and precautions.

†Methods for random sampling.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED
(Clause 2.3)

| LOT SIZE (1) | NUMBER OF CONTAINERS TO BE SELECTED (2) |
|-----------------|---|
| Up to 25 | 3 |
| 26 to 100 | 4 |
| 101 to 150 | 5 |
| 151 and above | 7 |

B-3. NUMBER OF TESTS

B-3.1 Test for determination of assay and ρ H shall be determined on individual containers and for the remaining other characteristics, tests shall be conducted on composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 For all those characteristics for which individual tests have been conducted, average (\bar{X}) and range (R) shall be calculated, range being the difference between the maximum and minimum of the test results and

$$\text{Average} = \frac{\text{sum of the test results}}{\text{number of tests}}$$

The lot shall be declared as conforming to the requirements of assay content if :

$\bar{X} - 0.6 R \geqslant$ the minimum value specified in Table 1 and for ρ H value the lot shall be declared as conforming to the specification if :

$$R/U-L \leqslant 0.9;$$

$$\bar{X} + 0.6 R \leqslant U; \text{ and}$$

$$\bar{X} - 0.6 R \geqslant L.$$

where U is upper specification limit and L is the lower specification limit.

B-4.2 For composite sample, the lot shall be declared as conforming to the requirements of this specification if it fails in none of the tests.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

| QUANTITY | UNIT | SYMBOL |
|---------------------------|----------|--------|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

Supplementary Units

| QUANTITY | UNIT | SYMBOL |
|-------------|-----------|--------|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

Derived Units

| QUANTITY | UNIT | SYMBOL | DEFINITION |
|----------------------|---------|--------|---------------------------------|
| Force | newton | N | 1 N = 1 kg.m/s ² |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 Wb/m ² |
| Frequency | hertz | Hz | 1 Hz = 1 c/s (s ⁻¹) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Electromotive force | volt | V | 1 V = 1 W/A |
| Pressure, stress | pascal | Pa | 1 Pa = 1 N/m ² |